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PATENT ABSTRACTS OF JAPAN

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(54) PRODUCTION OF GLASS SUBSTRATE FOR INFORMATION RECORDING MEDIUM

(57)Abstract:
PROBLEM TO BE SOLVED: To obtain a glass substrate for an information recording medium which suppresses the elution of alkali metal ions from the surface of the chemically tempered glass substrate and to obtain the information recording medium using the glass substrate and excellent in weather resistance.
SOLUTION: A glass substrate is chemically tempered by ion exchange in a tempering salt and then subjected to alkali removal treatment by immersion in a molten salt prepared by adding the nitrate of a third metl having a larger ionic radius than alkali metal ions in the glass to the tempering salt at a lower immersion temperature than the chemical tempering temperature. In the alkali ion removing step, the temperature of the glass substrate is room temperature to 400° C at the time when the substrate is immersed in the nitrate salt and the immersion time is ≥30 sec.

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CLAIMS

[Claim(s)]
[Claim 1] The manufacture approach of the glass substrate for information record media and the glass substrate for information record media which have the process which removes the alkali ion near the outermost front face in the ion-exchange layer on the front face of a substrate in the manufacture approach of the glass substrate for information record media by immersing the glass substrate by the ion exchange by which chemical-strengthening processing was carried out in an inorganic compound salt bath.
[Claim 2] The manufacture approach of the glass substrate for information record media according to claim 1 characterized by using the fused salt of a nitrate as an inorganic compound salt bath in the process which removes alkali ion.
[Claim 3] The manufacture approach of the glass substrate for information record media according to claim 1 characterized by using the nitrate of a metal with a larger ionic radius than sodium as an inorganic compound salt in the process which removes alkali ion.
[Claim 4] The manufacture approach of the glass substrate for information record media according to claim 1 or 2 characterized by for the temperature of the glass substrate at the time of being immersed in a nitrate being room temperature -400 degree C in the process which removes alkali ion, and immersion time amount being 30 seconds or more.

[Translation done.]

JP,A, 2000-293844

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] This invention relates to the information record medium which used this glass substrate for the manufacture approach of the glass substrate for information record media, and the list.
[0002]
[Description of the Prior Art] The substrate for information record media which was excellent in smooth nature and flatness for the improvement in recording density is needed with large-capacity-izing of an information recording device, and the very advantageous thing of a glass substrate ingredient is common especially as compared with the aluminium alloy and plastic material as other substrate ingredients for information record media. However, since glass is a brittle material, it is raising and using mechanical reinforcement for a surface by forming a chemical-strengthening layer in many cases.
[0003] However, it poses a problem that the rich layer of the alkali metal of a strengthening layer especially a potassium atom, or a sodium atom appears on a disk main front face in the substrate front face after chemical-strengthening processing of a glass substrate here. There is a danger that the migration of alkali-metal ion will cause reading incorrect actuation from this layer as the state of preservation of a disk and aging after subsequent information record-medium membrane formation because the carbonate and chloride of alkali metal deposit on a disk edge and the main front face. Moreover, alkali metal reacting with the record medium of a metal alloy, and causing incorrect actuation of equipment is also considered.
[0004] For this reason, after carrying out ion exchange treatment of the glass substrate front face, the alkali-metal ion of the maximum surface layer which is easy to be eluted is removed, and the dealkalization processing for raising chemical resistance and the cure against the closure of alkali-metal ion are performed. For example, there is an approach using an acid, especially strong acid like the approach of making the heated concentrated sulfuric acid contacting like the manufacture approach shown in a patent public presentation number and JP,10-226539,A. Moreover, like the manufacture approach shown in a patent public presentation number and JP,8-180402,A, into 80-100-degree C warm water, there is also a warm water art which carries out dealkalization metal ion processing of the glass substrate after ion exchange treatment by carrying out immersion processing for about 2 to 10 hours, in this case, impregnation processing of a divalent metallic ion is carried out to the maximum surface layer of the glass substrate after dealkalization metal ion processing, and the cure against the closure of alkali-metal ion is added further.
[0005]
[Problem(s) to be Solved by the Invention] however, in these dealkalization metal ion processings Although impregnation processing of a divalent metallic ion is required of the safety of using strong acid, such as heat concentrated sulfuric acid (it being heat concentrated sulfuric acid of 96% or more of concentration for example, at 100-degree-C **), and warm water processing as after treatment since [which needs immersion time amount at least 2 hours or more] there are few treatment effects the alkali-metal ion closure effectiveness — an alkali elution volume — 0.3-0.5microg/cm2 — it is — practically — chemical resistance — there is a problem of being unable to say that it is enough.
[0006] this invention — the manufacture approach of the glass substrate for information record media — setting — the alkali metal on the front face of glass after chemical-strengthening processing — in order to control the alkali migration from a rich layer, the alkali-metal ion of an after [a chemical strengthening] glass substrate front face is removed efficiently, and it is making into the technical problem to offer the information record medium excellent in the weatherability using the manufacture approach that the glass substrate excellent in chemical resistance is efficiently producible, and this glass substrate.
[0007]
[Means for Solving the Problem] this invention person is related with the manufacture approach of the glass substrate for information record media, as a result of examining the above-mentioned technical problem wholeheartedly. By the usual approach, in one-step strengthening, in two-step strengthening of a potassium nitrate, for example, the glass substrate which performed chemical-strengthening processing by the ion exchange at the temperature below the transition point of glass using the chemical-strengthening liquid of the inorganic compound salt which mixed the potassium nitrate and the sodium nitrate An inorganic compound salt with an again larger ionic radius than the alkali-metal ion in glass (sodium ion or lithium ion), for example, the melting temperature of the chemical-strengthening liquid of said inorganic compound salt which carried out little addition of potassium salt, a cesium salt, or the rubidium salt, and this chemical-strengthening liquid is set as temperature lower than the solution temperature at the time of the first chemical strengthening — it being alike, and, if immersed Thermal diffusion of the alkali metal on the front face of a glass substrate is carried out into this chemical-strengthening salt bath. Since the potassium ion by which little-among chemical-strengthening salt bath addition is carried out at this time, caesium ion, or rubidium ion has the large ionic radius, it is hard to be introduced all over the network of glass. That is, it could escape from the alkali metal which exists in the glass substrate surface layer easily, and a header and this invention were completed for the above-mentioned technical problem being attained by the glass surface layer excellent in chemical resistance being formed.
[0008] Namely, the manufacture approach of the glass substrate for information record media of this invention After an inorganic compound salt carries out chemical-strengthening processing of the glass substrate by the usual approach in the manufacture approach of the glass substrate for information record media, The chemical-strengthening salt bath of the inorganic compound salt which carried out little addition of the inorganic compound salt with a larger ionic radius than the alkali-metal ion in glass at the chemical-strengthening liquid which heated the glass substrate at room temperature -400 degree C, and used the glass substrate previously, It is alike and is immersed. the solution temperature of this chemical-strengthening salt bath is set as eye the 20-100-

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degree-C low rather than the solution temperature at the time of the first chemical strengthening — It is the dealkalization art which is characterized by diffusing only the alkali-metal ion on the front face of glass in this chemical-strengthening salt bath, and removes efficiently the alkali-metal ion on the front face of a glass substrate.
[0009]
[Embodiment of the Invention] Hereafter, this invention is explained to a detail.
[0010] In the manufacture approach of the glass substrate for information record media of this invention, it is immersed into the chemical-strengthening salt bath which heated the glass substrate, the ion and the ion exchange in a chemical-strengthening salt bath are carried out for the ion on the front face of glass, and the chemical strengthening of the glass substrate is carried out. This invention is processed for the glass substrate which carried out the chemical strengthening, and it is characterized by performing dealkalization processing.
[0011] As the approach of dealkalization processing, a glass substrate is beforehand made into room temperature -400 degree C. The chemical-strengthening salt bath of the inorganic compound salt which carried out little addition of the inorganic compound salt with a larger ionic radius than the alkali-metal ion in glass into the chemical-strengthening salt bath of the inorganic compound salt which used the glass substrate previously, The solution temperature of this chemical-strengthening salt bath is preferably immersed in this chemical-strengthening salt bath set as eye the 20-100-degree-C low rather than the solution temperature at the time of the first chemical strengthening for 5 - 30 minutes 30 seconds or more. The glass substrate temperature at the time of immersion should just be beyond a room temperature at this chemical-strengthening salt bath. A reaction with a glass substrate becomes that the temperature of a glass substrate is 100 degrees C or more early preferably, the processing time can be managed in a short time, and productive efficiency improves. Moreover, since deformation of a glass substrate occurs at the immersion temperature of 500 degrees C or more, a productive-efficiency top is not good. Moreover, dealkalization effectiveness with the processing time sufficient in 30 or less seconds is hard to be acquired. Thus, the sodium ion near [the] a glass front face, potassium ion, or a lithium ion emits the glass substrate processed with this chemical-strengthening salt bath into this chemical-strengthening salt bath by thermal diffusion, and it serves as elevated-temperature dealkalization processing. Moreover, this chemical-strengthening salt crystal object adhering to the substrate front face after processing can be easily dissolved with distilled water, and a pure substrate front face is obtained.
[0012] Moreover, in this invention, for example, aluminosilicate system glass, soda lime glass, borosilicate glass, alumino borosilicate glass, etc. can be used as what can form a strengthening layer by chemical-strengthening processing as a glass ingredient.
[0013] In addition, the basic principle of the dealkalization approach of this invention is an ionic diffusion by the concentration difference of the metal ion in fused salt, and the alkali-metal ion in glass. The nitrate as third mineral salt compound with a larger ionic radius than the alkali-metal ion in glass is added in the chemical-strengthening liquid which performed ion exchange treatment on introduction usual temperature conditions, and was used for ion exchange treatment after that. Although the alkali-metal ion in glass will carry out an ionic diffusion and it will slip out of it out of glass if immersed in the fused salt bath made into temperature conditions somewhat lower than the first ion-exchange-treatment temperature Then Since the ion (potassium ion, a lithium ion, caesium ion, rubidium ion, etc.) of fused salt has little heat energy required to carry out an ionic diffusion into glass, An ionic diffusion is carried out and it is hard to enter into glass, and on the other hand, there will be more alkali-metal ion (sodium ion, lithium ion) which falls out out of glass on a target, and comes out, and, as for the income-and-outgo balance of mutual ion, the dealkalization phenomenon on the front face of glass will be performed as a result. This principle can be discovered based on many experiments and theoretical considerations, and results in completion of this invention. As long as it says from this principle, to the glass which contains neither a lithium nor a potassium, a salt like a potassium nitrate is [that what is necessary is just a metal with a larger ionic radius than the alkali metal in glass] sufficient as the nitrate as third mineral salt compound to be used. Moreover, a metal salt like a silver nitrate is sufficient.
[0014]
[Example] (Example 1) After carrying out chemical-strengthening processing of the alumino silicate glass substrate in the 420-degree C fused salt bath of a potassium nitrate for 4 hours and washing out an adhering deposit salt with water, predetermined time immersion was carried out at the salt bath which added the nitric-acid rubidium 10% during the 350-degree C fused salt bath of a potassium nitrate, and the alumino silicate glass substrate was taken out from the salt bath after that, and was cooled radiationally. Distilled water washed after radiationnal cooling and measurement of the alkali-metal (Na, Li, K) concentration which performed the waterproof trial of a glass substrate and was eluted from glass in this sample was measured with atomic-absorption-analysis equipment. In addition, the waterproof trial was immersed into the Teflon beaker which filled the glass substrate with 50ml distilled water, moved the Teflon beaker containing the glass substrate to the constant temperature bath kept at 80 degrees C, and carried out quantitative analysis of the amount of alkali metal which held for 24 hours and was eluted from the glass substrate in 80-degree C warm water with the atomic absorption method. The result was as being shown in Table 1.
[0015]
表１．ガラス基板のアルカリ溶出量（μg／cm²）

	処理時間（分）			
	未処理	５分	１５分	３０分
Ｎａ	0.350	0.090	0.058	0.046
Ｌｉ	0.075	0.008	0.008	0.008
Ｋ	0.158	0.017	0.000	0.000
合計	0.583	0.115	0.066	0.054

As shown in Table 1, the alkali elution volume of a glass substrate decreased to unsettled 1/4 in 5 minutes of processing times, and it was checked that dealkalization processing is performed for a short time.
[0016] (Example 2) Next, after carrying out chemical-strengthening processing of the same glass substrate as an example 1 similarly and washing out an adhering deposit salt with water, predetermined time immersion was carried out at the salt bath which added the silver nitrate 10% during the 380-degree C fused salt bath of a potassium nitrate, and the glass substrate was taken out from the salt bath after that, and was cooled radiationally. Distilled water washed after radiationnal cooling and measurement of the alkali-metal ion (Na, Li, K) concentration which performed the waterproof trial of a glass substrate and was eluted from glass in this sample was measured with atomic-absorption-analysis equipment. The result became as shown in Table 2.

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JP. A, 2000-293844

[0017]

表２．ガラス基板のアルカリ溶出量（μｇ／ｃｍ²）

	処理時間（分）				
	未処理	３０秒	１分	５分	１０分
Ｎａ	0.315	0.035	0.034	0.011	0.008
Ｌｉ	0.007	0.000	0.000	0.003	0.026
Ｋ	0.120	0.110	0.110	0.120	0.140
合計	0.442	0.144	0.144	0.134	0.174

As shown in Table 2, even when a silver nitrate was added to potassium-nitrate chemical-strengthening fused salt, the alkali elution volume of a glass substrate decreased to unsettled 1/3 in 30 seconds of processing times, and it was checked that dealkalization processing is performed extremely for a short time.

[0018]

[Effect of the Invention] As explained above, the glass substrate manufactured by carrying out dealkalization processing according to the manufacture approach of the glass substrate for information record media of this invention can control remarkably the alkali migration on the front face of a glass substrate, and can raise the dependability of an information record medium by leaps and bounds by the ability of the glass substrate which does not have a bad influence on the information record medium formed on a glass substrate to be offered.

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TECHNICAL FIELD

[Field of the Invention] This invention relates to the information record medium which used this glass substrate for the manufacture approach of the glass substrate for information record media, and the list.

[Translation done.]

PRIOR ART

[Description of the Prior Art] The substrate for information record media which was excellent in smooth nature and flatness for the improvement in recording density is needed with large-capacity-izing of an information recording device, and the very advantageous thing of a glass substrate ingredient is common especially as compared with the aluminium alloy and plastic material as other substrate ingredients for information record media. However, since glass is a brittle material, it is raising and using mechanical reinforcement for a surface by forming a chemical-strengthening layer in many cases.
[0003] However, it poses a problem that the rich layer of the alkali metal of a strengthening layer especially a potassium atom, or a sodium atom appears on a disk main front face in the substrate front face after chemical-strengthening processing of a glass substrate here. There is a danger that the migration of alkali-metal ion will cause reading incorrect actuation from this layer as the state of preservation of a disk and aging after subsequent information record-medium membrane formation because the carbonate and chloride of alkali metal deposit on a disk edge and the main front face. Moreover, alkali metal reacting with the record medium of a metal alloy, and causing incorrect actuation of equipment is also considered.
[0004] For this reason, after carrying out ion exchange treatment of the glass substrate front face, the alkali-metal ion of the maximum surface layer which is easy to be eluted is removed, and the dealkalization processing for raising chemical resistance and the cure against the closure of alkali-metal ion are performed. For example, there is an approach using an acid, especially strong acid like the approach of making the heated concentrated sulfuric acid contacting like the manufacture approach shown in a patent public presentation number and JP,10-226539,A. Moreover, like the manufacture approach shown in a patent public presentation number and JP,8-180402,A, into 80-100-degree C warm water, there is also a warm water art which carries out dealkalization metal ion processing of the glass substrate after ion exchange treatment by carrying out immersion processing for about 2 to 10 hours, in this case, impregnation processing of a divalent metallic ion is carried out to the maximum surface layer of the glass substrate after dealkalization metal ion processing, and the cure against the closure of alkali-metal ion is added further.

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JP, A, 2000-293844

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EFFECT OF THE INVENTION

[Effect of the Invention] As explained above, the glass substrate manufactured by carrying out dealkalization processing according to the manufacture approach of the glass substrate for information record media of this invention can control remarkably the alkali migration on the front face of a glass substrate, and can raise the dependability of an information record medium by leaps and bounds by the ability of the glass substrate which does not have a bad influence on the information record medium formed on a glass substrate to be offered.

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TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] however, in these dealkalization metal ion processings Although impregnation processing of a divalent metallic ion is required of the safety of using strong acid, such as heat concentrated sulfuric acid (it being heat concentrated sulfuric acid of 96% or more of concentration for example, at 100-degree-C **), and warm water processing as after treatment since [which needs immersion time amount at least 2 hours or more] there are few treatment effects the alkali-metal ion closure effectiveness — an alkali elution volume — 0.3-0.5microg/cm2 — it is — practically — chemical resistance — there is a problem of being unable to say that it is enough.
[0006] this invention — the manufacture approach of the glass substrate for information record media — setting — the alkali metal on the front face of glass after chemical-strengthening processing — in order to control the alkali migration from a rich layer, the alkali-metal ion of an after [a chemical strengthening] glass substrate front face is removed efficiently, and it is making into the technical problem to offer the information record medium excellent in the weatherability using the manufacture approach that the glass substrate excellent in chemical resistance is efficiently producible, and this glass substrate.

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MEANS

[Means for Solving the Problem] this invention person is related with the manufacture approach of the glass substrate for information record media, as a result of examining the above-mentioned technical problem wholeheartedly. By the usual approach, in one-step strengthening, in two-step strengthening of a potassium nitrate, for example, the glass substrate which performed chemical-strengthening processing by the ion exchange at the temperature below the transition point of glass using the chemical-strengthening liquid of the inorganic compound salt which mixed the potassium nitrate and the sodium nitrate An inorganic compound salt with an again larger ionic radius than the alkali-metal ion in glass (sodium ion or lithium ion), for example, the melting temperature of the chemical-strengthening liquid of said inorganic compound salt which carried out little addition of potassium salt, a cesium salt, or the rubidium salt, and this chemical-strengthening liquid is set as temperature lower than the solution temperature at the time of the first chemical strengthening — it being alike, and, if immersed Thermal diffusion of the alkali metal on the front face of a glass substrate is carried out into this chemical-strengthening salt bath. Since the potassium ion by which little-among chemical-strengthening salt bath addition is carried out at this time, caesium ion, or rubidium ion has the large ionic radius, it is hard to be introduced all over the network of glass. That is, it could escape from the alkali metal which exists in the glass substrate surface layer easily, and a header and this invention were completed for the above-mentioned technical problem being attained by the glass surface layer excellent in chemical resistance being formed.
[0008] Namely, the manufacture approach of the glass substrate for information record media of this invention After an inorganic compound salt carries out chemical-strengthening processing of the glass substrate by the usual approach in the manufacture approach of the glass substrate for information record media, The chemical-strengthening salt bath of the inorganic compound salt which carried out little addition of the inorganic compound salt with a larger ionic radius than the alkali-metal ion in glass at the chemical-strengthening liquid which heated the glass substrate at room temperature -400 degree C, and used the glass substrate previously, It is alike and is immersed. the solution temperature of this chemical-strengthening salt bath is set as eye the 20-100-degree-C low rather than the solution temperature at the time of the first chemical strengthening — It is the dealkalization art which is characterized by diffusing only the alkali-metal ion on the front face of glass in this chemical-strengthening salt bath, and removes efficiently the alkali-metal ion on the front face of a glass substrate.
[0009]
[Embodiment of the Invention] Hereafter, this invention is explained to a detail.
[0010] In the manufacture approach of the glass substrate for information record media of this invention, it is immersed into the chemical-strengthening salt bath which heated the glass substrate, the ion and the ion exchange in a chemical-strengthening salt bath are carried out for the ion on the front face of glass, and the chemical strengthening of the glass substrate is carried out. This invention is processed for the glass substrate which carried out the chemical strengthening, and it is characterized by performing dealkalization processing.
[0011] As the approach of dealkalization processing, a glass substrate is beforehand made into room temperature -400 degree C. The chemical-strengthening salt bath of the inorganic compound salt which carried out little addition of the inorganic compound salt with a larger ionic radius than the alkali-metal ion in glass into the chemical-strengthening salt bath of the inorganic compound salt which used the glass substrate previously, The solution temperature of this chemical-strengthening salt bath is preferably immersed in this chemical-strengthening salt bath set as eye the 20-100-degree-C low rather than the solution temperature at the time of the first chemical strengthening for 5 - 30 minutes 30 seconds or more. The glass substrate temperature at the time of immersion should just be beyond a room temperature at this chemical-strengthening salt bath. A reaction with a glass substrate becomes that the temperature of a glass substrate is 100 degrees C or more early preferably, the processing time can be managed in a short time, and productive efficiency improves. Moreover, since deformation of a glass substrate occurs at the immersion temperature of 500 degrees C or more, a productive-efficiency top is not good. Moreover, dealkalization effectiveness with the processing time sufficient in 30 or less seconds is hard to be acquired. Thus, the sodium ion near [the] a glass front face, potassium ion, or a lithium ion emits the glass substrate processed with this chemical-strengthening salt bath into this chemical-strengthening salt bath by thermal diffusion, and it serves as elevated-temperature dealkalization processing. Moreover, this chemical-strengthening salt crystal object adhering to the substrate front face after processing can be easily dissolved with distilled water, and a pure substrate front face is obtained.
[0012] Moreover, in this invention, for example, aluminosilicate system glass, soda lime glass, borosilicate glass, alumino borosilicate glass, etc. can be used as what can form a strengthening layer by chemical-strengthening processing as a glass ingredient.
[0013] In addition, the basic principle of the dealkalization approach of this invention is an ionic diffusion by the concentration difference of the metal ion in fused salt, and the alkali-metal ion in glass. The nitrate as third mineral salt compound with a larger ionic radius than the alkali-metal ion in glass is added in the chemical-strengthening liquid which performed ion exchange treatment on introduction usual temperature conditions, and was used for ion exchange treatment after that. Although the alkali-metal ion in glass will carry out an ionic diffusion and it will slip out of it out of glass if immersed in the fused salt bath made into temperature conditions somewhat lower than the first ion-exchange-treatment temperature Then Since the ion (potassium ion, a lithium ion, caesium ion, rubidium ion, etc.) of fused salt has little heat energy required to carry out an ionic diffusion into glass, An ionic diffusion is carried out and it is hard to enter into glass, and on the other hand, there will be more alkali-metal ion (sodium ion, lithium ion) which falls out out of glass on a target, and comes out, and, as for the income-and-outgo balance of mutual ion, the dealkalization phenomenon on the front face of glass will be performed as a result. This principle can be discovered based on many experiments and theoretical considerations, and results in completion of this invention. As long as it says from this principle, to the

JP,A, 2000-293844

glass which contains neither a lithium nor a potassium, a salt like a potassium nitrate is [that what is necessary is just a metal with a larger ionic radius than the alkali metal in glass] sufficient as the nitrate as third mineral salt compound to be used. Moreover, a metal salt like a silver nitrate is sufficient.

[Translation done.]

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EXAMPLE

[Example] (Example 1) After carrying out chemical-strengthening processing of the alumino silicate glass substrate in the 420-degree C fused salt bath of a potassium nitrate for 4 hours and washing out an adhering deposit salt with water, predetermined time immersion was carried out at the salt bath which added the nitric-acid rubidium 10% during the 350-degree C fused salt bath of a potassium nitrate, and the alumino silicate glass substrate was taken out from the salt bath after that, and was cooled radiationally. Distilled water washed after radiationnal cooling and measurement of the alkali-metal (Na, Li, K) concentration which performed the waterproof trial of a glass substrate and was eluted from glass in this sample was measured with atomic-absorption-analysis equipment. In addition, the waterproof trial was immersed into the Teflon beaker which filled the glass substrate with 50ml distilled water, moved the Teflon beaker containing the glass substrate to the constant temperature bath kept at 80 degrees C, and carried out quantitative analysis of the amount of alkali metal which held for 24 hours and was eluted from the glass substrate in 80-degree C warm water with the atomic absorption method. The result was as being shown in Table 1.
[0015]
表１．ガラス基板のアルカリ溶出量（$\mu g/cm^2$）

	処理時間（分）			
	未処理	５分	１５分	３０分
Na	0.350	0.090	0.058	0.046
Li	0.075	0.008	0.008	0.008
K	0.158	0.017	0.000	0.000
合計	0.583	0.115	0.066	0.054

As shown in Table 1, the alkali elution volume of a glass substrate decreased to unsettled 1/4 in 5 minutes of processing times, and it was checked that dealkalization processing is performed for a short time.
[0016] (Example 2) Next, after carrying out chemical-strengthening processing of the same glass substrate as an example 1 similarly and washing out an adhering deposit salt with water, predetermined time immersion was carried out at the salt bath which added the silver nitrate 10% during the 380-degree C fused salt bath of a potassium nitrate, and the glass substrate was taken out from the salt bath after that, and was cooled radiationally. Distilled water washed after radiationnal cooling and measurement of the alkali-metal ion (Na, Li, K) concentration which performed the waterproof trial of a glass substrate and was eluted from glass in this sample was measured with atomic-absorption-analysis equipment. The result became as shown in Table 2.
[0017]
表２．ガラス基板のアルカリ溶出量（$\mu g/cm^2$）

	処理時間（分）				
	未処理	３０秒	１分	５分	１０分
Na	0.315	0.035	0.034	0.011	0.008
Li	0.007	0.000	0.000	0.003	0.026
K	0.120	0.110	0.110	0.120	0.140
合計	0.442	0.144	0.144	0.134	0.174

As shown in Table 2, even when a silver nitrate was added to potassium-nitrate chemical-strengthening fused salt, the alkali elution volume of a glass substrate decreased to unsettled 1/3 in 30 seconds of processing times, and it was checked that dealkalization processing is performed extremely for a short time.

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(54)【発明の名称】 情報記録媒体用ガラス基板の製造方法

(57)【要約】
【課題】化学強化したガラス基板表面からのアルカリ金属イオンの溶出を抑制した情報記録媒体用ガラス基板及びこのガラス基板を用いた耐候性に優れた情報記録媒体を提供する。
【解決手段】情報記録媒体用ガラス基板の製造方法において、イオン交換による化学強化処理を行った後、強化液にガラス中のアルカリ金属イオンよりイオン半径の大きい第三の硝酸塩を添加した溶融塩に浸漬して化学強化より低い温度で浸漬することで脱アルカリ処理ができる。

1

【特許請求の範囲】

【請求項１】情報記録媒体用ガラス基板の製造方法において、イオン交換による化学強化処理されたガラス基板を、無機化合物塩浴に浸漬することにより、基板表面のイオン交換層の中、最外表面近傍のアルカリイオンを除去する工程を有する情報記録媒体用ガラス基板の製造方法および情報記録媒体用ガラス基板。

【請求項２】アルカリイオンを除去する工程において、無機化合物塩浴として硝酸塩の溶融塩を用いることを特徴とする請求項1記載の情報記録媒体用ガラス基板の製造方法。

【請求項３】アルカリイオンを除去する工程において、無機化合物塩としてナトリウムよりイオン半径の大きい金属の硝酸塩を用いることを特徴とする請求項1記載の情報記録媒体用ガラス基板の製造方法。

【請求項４】アルカリイオンを除去する工程において、硝酸塩に浸漬する際のガラス基板の温度が室温～４００℃であり、浸漬時間が３０秒以上であることを特徴とする請求項１又は請求項２記載の情報記録媒体用ガラス基板の製造方法。

【発明の詳細な説明】

【０００１】

【発明の属する技術分野】本発明は情報記録媒体用ガラス基板の製造方法、並びにこのガラス基板を用いた情報記録媒体に関するものである。

【０００２】

【従来の技術】情報記録装置の大容量化にともなって、記録密度向上のために平滑性、平面度の優れた情報記録媒体用基板が必要とされ、中でもガラス基板材料は他の情報記録媒体用基板材料としてのアルミニウム合金やプラスチック材料に比較して非常に有利であることが一般的である。しかし、ガラスは脆性材料であることから表層に化学強化層を形成することで機械的な強度を上げて使用している場合が多い。

【０００３】しかし、ここで問題となるのはガラス基板の化学強化処理後の基板表面に強化層のアルカリ金属、特にカリウム原子、またはナトリウム原子のリッチな層がディスク主表面に現れることである。この層からアルカリ金属イオンのマイグレーションにより、ディスクの保存状態や、その後の情報記録媒体成膜後の経時変化としてディスク端部、主表面にアルカリ金属の炭酸塩や塩化物が析出することで、読み取り誤作動を引き起こす危険性が有る。また、アルカリ金属が金属合金の記録媒体と反応して、装置の誤作動を引き起こすことも考えられる。

【０００４】このため、ガラス基板表面をイオン交換処理した後、溶出し易い最表面層のアルカリ金属イオンを除去し、耐化学性を上げるための脱アルカリ処理やアルカリ金属イオンの封止対策が行われている。例えば、特許公開番号、特開平１０－２２６５３９に示される製造

2

方法のように、加熱した濃硫酸と接触させる方法のように酸、特に強酸を用いる方法がある。また特許公開番号、特開平８－１８０４０２に示される製造方法のように、８０～１００℃の温水中に、イオン交換処理後のガラス基板を２～１０時間程度浸漬処理することで脱アルカリ金属イオン処理する温水処理方法もあり、この場合更に、脱アルカリ金属イオン処理後ガラス基板の最表面層に対して２価金属イオンの注入処理をして、アルカリ金属イオンの封止対策が加えられている。

【０００５】

【発明が解決しようとする課題】しかし、これらの脱アルカリ金属イオン処理では、熱濃硫酸（例えば、１００℃超で濃度９６％以上の熱濃硫酸）等の強酸を使用することの安全性や、温水処理では少なくとも２時間以上の浸漬時間が必要であり、また処理効果が少ないため後処理として２価金属イオンの注入処理が必要であるが、そのアルカリ金属イオン封止効果は、アルカリ溶出量が０．３～０．５μg／cm² で、実用上耐化学性充分とは言えない等の問題がある。

【０００６】本発明は、情報記録媒体用ガラス基板の製造方法において、化学強化処理後のガラス表面のアルカリ金属リッチな層からのアルカリマイグレーションを抑制するため、化学強化後ガラス基板表面のアルカリ金属イオンを効率良く除去し、耐化学性に優れたガラス基板を効率良く生産できる製造方法及びこのガラス基板を用いた耐候性に優れた情報記録媒体を提供することを課題としている。

【０００７】

【課題を解決するための手段】本発明者は、上記の課題について鋭意検討した結果、情報記録媒体用ガラス基板の製造方法に関し、通常の方法で例えば１段強化の場合は硝酸カリウムを、２段強化の場合は硝酸カリウムと硝酸ナトリウムを混合した無機化合物塩の化学強化液を用いてガラスの転移点以下の温度でイオン交換による化学強化処理を行ったガラス基板を、再びガラス中のアルカリ金属イオン（ナトリウムイオンあるいはリチウムイオン）よりイオン半径の大きい無機化合物塩、例えばカリウム塩、セシウム塩、あるいはルビジウム塩等を少量添加した前記無機化合物塩の化学強化液、該化学強化液の溶融温度は最初の化学強化時の液温よりも低い温度に設定してある、に浸漬すると、ガラス基板表面のアルカリ金属は該化学強化塩浴中に熱拡散する。この時化学強化塩浴中少量添加されているカリウムイオン、セシウムイオン、あるいはルビジウムイオンはイオン半径が大きいため、ガラスのネットワーク中には導入されにくい。つまり、容易にガラス基板表面層に存在しているアルカリ金属が抜け取れ、耐化学性に優れたガラス表面層が形成されることで、上記の課題が達成されることを見出し、本発明を完成させた。

【０００８】即ち、本発明の情報記録媒体用ガラス基板

の製造方法は、情報記録媒体用ガラス基板の製造方法において、ガラス基板を通常の方法で無機化合物塩の化学強化処理した後、ガラス基板を室温～４００℃に加熱し、そのガラス基板を先に使用した化学強化液にガラス中のアルカリ金属イオンよりイオン半径の大きい無機化合物塩を少量添加した無機化合物塩の化学強化塩浴、該化学強化塩浴の液温は最初の化学強化時の液温よりも２０～１００℃低目に設定してある、に浸漬して、ガラス表面のアルカリ金属イオンのみ該化学強化塩浴中に拡散させることを特徴とし、ガラス基板表面のアルカリ金属イオンを効率良く除去する脱アルカリ処理方法である。

【０００９】

【発明の実施の形態】以下、本発明を詳細に説明する。

【００１０】本発明の情報記録媒体用ガラス基板の製造方法においては、ガラス基板を加熱した化学強化塩浴中に浸漬し、ガラス表面のイオンを化学強化塩浴中のイオンとイオン交換をして、ガラス基板を化学強化する。その化学強化したガラス基板を本発明の処理を行い、脱アルカリ処理を行うことを特徴とする。

【００１１】脱アルカリ処理の方法としては、ガラス基板を予め室温～４００℃にし、そのガラス基板を、先に使用した無機化合物塩の化学強化塩浴中にガラス中のアルカリ金属イオンよりイオン半径の大きい無機化合物塩を少量添加した無機化合物塩の化学強化塩浴、該化学強化塩浴の液温は最初の化学強化時の液温よりも２０～１００℃低目に設定してある、該化学強化塩浴に３０秒以上好ましくは５～３０分間浸漬する。該化学強化塩浴に浸漬時のガラス基板温度は、室温以上であれば良い。好ましくは、ガラス基板の温度が１００℃以上であると、ガラス基板との反応が早くなり、処理時間が短時間で済み、生産効率が良化する。また、浸漬温度５００℃以上ではガラス基板の変形が発生するため、生産効率上良くない。また処理時間が３０秒以下では充分な脱アルカリ効果が得られにくい。このように該化学強化塩浴で処理されたガラス基板は、そのガラス表面付近のナトリウムイオン、カリウムイオン、あるいはリチウムイオンが熱拡散により、該化学強化塩浴中に放出し、高温脱アルカリ処理となる。また、処理後基板表面に付着した該化学強化塩結晶物は蒸留水で容易に溶解することができ、清浄な基板表面が得られる。

【００１２】また本発明において、ガラス材料としては化学強化処理で強化層を形成することのできるものとして、例えばアルミノシリケート系ガラス、ソーダライム＊

＊ガラス、ホウケイ酸ガラス、アルミノホウケイ酸ガラス等を用いることができる。

【００１３】なお、本発明の脱アルカリ方法の基本原理は、溶融塩中の金属イオンとガラス中のアルカリ金属イオンの濃度差による、イオン拡散である。初めに通常の温度条件でイオン交換処理を行い、その後イオン交換処理に用いた化学強化液にガラス中のアルカリ金属イオンよりイオン半径が大きい第三の無機塩化合物としての硝酸塩を添加し、初めのイオン交換処理温度より少し低い温度条件にした溶融塩浴に浸漬すると、ガラス中のアルカリ金属イオンはガラス中からイオン拡散して抜け出すが、その時溶融塩のイオン（カリウムイオン、リチウムイオン、セシウムイオン、ルビジウムイオン等）はガラス中にイオン拡散していくに必要な熱エネルギーが少ないため、ガラス中にイオン拡散して入り込み難く、相互のイオンの収支バランスは一方的にガラス中から抜け出てくるアルカリ金属イオン（ナトリウムイオン、リチウムイオン）の方が多く、結果的にガラス表面の脱アルカリ現象が行われていることになる。この原理は幾多の実験や理論的考察に基づき発見できたものであり、この発明の完成に到ったものである。この原理から言えば、用いる第三の無機塩化合物としての硝酸塩は、ガラス中のアルカリ金属よりイオン半径の大きい金属であれば良く、リチウムやカリウムを含まないガラスに対しては硝酸カリウムのような塩でも良い。また、硝酸銀のような金属塩でもよい。

【００１４】

【実施例】（実施例１）アルミノシリケートガラス基板を４２０℃の硝酸カリウムの溶融塩浴中で４時間化学強化処理し、付着している析出塩を水で洗い落とした後、そのアルミノシリケートガラス基板を３５０℃の硝酸カリウムの溶融塩浴中に１０％硝酸ルビジウムを添加した塩浴に所定時間浸漬し、その後塩浴から取りだし、放冷した。放冷後、蒸留水で洗浄し、このサンプルをガラス基板の耐水性試験を行い、ガラスから溶出したアルカリ金属（Ｎａ、Ｌｉ、Ｋ）濃度の測定を原子吸光分析装置にて測定した。なお耐水性試験は、ガラス基板を５０ｍｌの蒸留水で満たしたテフロンビーカー中に浸漬し、そのガラス基板の入ったテフロンビーカーを８０℃に保った恒温水槽に移し、８０℃の温水中に２４時間保持してガラス基板から溶出したアルカリ金属量を原子吸光法で定量分析した。その結果は表１に示す通りであった。

【００１５】

表１．ガラス基板のアルカリ溶出量（μｇ／cm²）

	処理時間（分）			
	未処理	５分	１５分	３０分
Ｎａ	0.350	0.090	0.058	0.046
Ｌｉ	0.075	0.008	0.008	0.008
Ｋ	0.158	0.017	0.000	0.000
合計	0.583	0.115	0.066	0.054

表１に示したように、ガラス基板のアルカリ溶出量は処理時間５分で未処理の１／４に減少し、短時間で脱アルカリ処理が行なわれていることが確認された。

【００１６】（実施例２）次に、実施例1と同じガラス基板を同様に化学強化処理し、付着している析出塩を水で洗い落とした後、そのガラス基板を３８０℃の硝酸カリウムの溶融塩浴中に１０％硝酸銀を添加した塩浴に所定時間浸漬し、その後塩浴から取りだし、放冷した。放冷後、蒸留水で洗浄し、このサンプルをガラス基板の耐水性試験を行い、ガラスから溶出したアルカリ金属イオン（Ｎａ、Ｌｉ、Ｋ）濃度の測定を原子吸光分析装置にて測定した。その結果は表２に示す通りになった。

【００１７】

表２．ガラス基板のアルカリ溶出量（$\mu g/cm^2$）

	処理時間（分）				
	未処理	３０秒	１分	５分	１０分
Ｎａ	0.315	0.035	0.034	0.011	0.008
Ｌｉ	0.007	0.000	0.000	0.003	0.026
Ｋ	0.120	0.110	0.110	0.120	0.140
合計	0.442	0.144	0.144	0.134	0.174

表２に示したように、硝酸カリウム化学強化溶融塩に硝酸銀を添加した場合でも、ガラス基板のアルカリ溶出量は処理時間３０秒で未処理の１／３に減少し、極めて短時間で脱アルカリ処理が行なわれていることが確認された。

【００１８】

【発明の効果】以上説明したように、本発明の情報記録媒体用ガラス基板の製造方法に従って脱アルカリ処理をして製造されたガラス基板は、ガラス基板表面のアルカリマイグレーションを著しく抑制し、ガラス基板上に成膜される情報記録媒体に悪影響を及ぼさないガラス基板を提供できることで、情報記録媒体の信頼性を飛躍的に向上させることができるものである。